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TRANSMISSION ELECTRON MICROSCOPY STUDY OF ALUMINA

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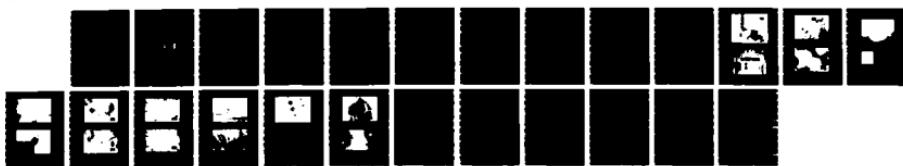
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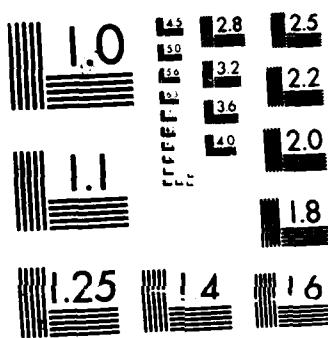
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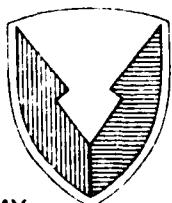
TRANSMISSION ELECTRON MICROSCOPY STUDY OF ALUMINA FIBER-REINFORCED MAGNESIUM COMPOSITE

ERNEST SUNG CHOR CHIN
METALS RESEARCH DIVISION

January 1987

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ABSTRACT

 A detailed microstructural analysis of alumina (FP) fiber-reinforced ZE41A magnesium composite was performed by means of transmission electron microscopy (TEM). Fifty-five volume percent FP fiber-reinforced ZE41A magnesium composite thin foils were prepared by methods of mechanical polishing and ion thinning. Three major areas of interest in the composite microstructure were studied; the filaments, the interface (fiber/matrix reaction zone), and the matrix. In addition, the effects of heat treatments at 450°C on the composite microstructures were investigated.

It was found that the alumina/magnesium interface consisted of a thin layer of $MgAl_2O_4$ spinel and relatively large magnesium oxide (MgO) particles. A concentric layer of magnesium alloy grains encircling the alumina filaments was also observed. MgZn¹ precipitates were also identified within the magnesium matrix microstructures. Upon heat treatment at 450°C for 95 hours, polygonization of the magnesium matrix occurred while the spinel interfacial layer grew with decreasing magnesium oxide particle size.

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INTRODUCTION

Metal matrix composites are some of the most exciting materials being studied today. The potential of tailoring composite properties for specific applications is enormous and beneficial to both current and future Army material needs. Because of this, numerous metal matrix composite systems are being evaluated by the Army for high stiffness, low density, and wear applications.¹

Currently, the knowledge of the strengthening mechanisms operating in metal matrix composites is limited. The role of the fiber/matrix interface is still poorly understood. It remains in doubt whether or not optimal properties have been achieved in today's composite systems. This uncertainty is one of the many factors that has hindered design engineers from implementing metal matrix composites in critical load bearing applications.

The object of this research effort is to gain a better understanding of the microstructural aspects in metal matrix composites. The role of each component (the fiber, the interface, and the matrix) in contributing to the bulk composite properties will be explored. The results and information gained from this study will contribute to the improvements in composite fabrication processes as well as in post heat treatments so as to optimize the overall properties of metal matrix composites.

The as-received alumina/ZE41A Mg composites used for this research effort were unidirectional fiber-reinforced composite plates. The 55 volume percent FP/Mg plates were in the as-cast condition and, in general, representative of the state-of-the-art material commercially available from DuPont.

MATERIAL BACKGROUND

The composite system studied in this research effort is the DuPont product FP (α -Al₂O₃)/ZE41A Mg. The FP/ZE41A Mg is unidirectionally reinforced with continuous FP (alumina) filaments and is commercially available from E. I. DuPont DeNemours and Company.

Fiber FP is the trade name for DuPont's continuous polycrystalline α -Al₂O₃ filament. ² The FP fibers are 99% pure and have a density equal to 99% of the theoretical value. ² The mechanical behavior of this reinforcement has been well characterized by Nunes.³

The matrix material is the ZE41A magnesium casting alloy which has the following chemical composition (see Table 1).⁴

FP/ZE41A Mg composites are fabricated by means of a molten liquid metal infiltration process.² This process has made possible the casting of small complex-shaped composite components as well as the fabrication of test plates. The molten metal infiltration process is currently being scaled-up to cast relatively large and complex composite components for both military and commercial applications.

1. LIVITT, A. P. *Army Metal Matrix Composite Program Overview*. U.S. Army Materials Technology Laboratory, AMMRC SP 83-3, April 1983.
2. DHINGRA, A. K., and KRUEGER, W. H. *New Engineering Material-Magnesium Castings Reinforced With DuPont Continuous Alumina Fiber FP*. E. I. DuPont DeNemours and Company, Experimental Station, Wilmington, Delaware, Presented at the 36th World Conference on Magnesium, Oslo, Norway, June 25-26 1979.
3. NUNES, J. *Tensile Property Evaluation of Polycrystalline Alumina Filaments and Their Properties*. U.S. Army Materials Technology Laboratory, AMMRC TR 82-61, December 1982.
4. Properties and Selection: Nonferrous Alloys and Pure Metals. *METALS HANDBOOK* Ninth Edition, v. 2, ASM, 1979.

TABLE 1. CHEMICAL COMPOSITION OF ZE41A MAGNESIUM CASTING ALLOY

Weight Percent							
Zn	Rare Earth (as misch metal)	Zr	Mn	Cu	Ni	Others	Rem.
3.5 to 5.0	0.75 to 1.75	0.40 to 1.0	0.15 max	0.10 max	0.01 max	0.30 max	Mg

SPECIMEN PREPARATION AND EXPERIMENTAL PROCEDURE

Transmission electron microscopy was the analytical tool used to evaluate the metal matrix composite microstructure in this study. In order to perform such analyses by means of transmission electron microscopy, thin foil specimens had to be prepared. Due to the mutually opposing characteristics of the composite components, namely the inert and wear resisting properties of the ceramic fibers and the relative reactive and the softness of the magnesium matrix, it is extremely difficult to prepare both flat and thin composite foils. The specimen thinning process developed for metal matrix composites is tedious and time consuming, and will be described below.

Prior to specimen thinning, the as-received FP/ZE41A Mg plates were cut and wafered in the direction perpendicular to the fiber orientation. Using a diamond blade, composite slices of approximately 0.5 mm thick were obtained. The thin slices were then mounted on a flat block (or the Gatan model 623 disc grinder) with double sided tape. Mounting can also be done using an acetone soluble cement. The specimens were then hand polished through a sequence of 200, 300, 400, and 600 grit silicon carbide papers and a series of 6 μm , 3 μm , and 1 μm diamond pastes on glass plates. A composite foil 50 μm thick was obtained through this process. An attempt was made to further thin the composite foil by means of jet electropolishing. However, due to the preferential attack on the matrix material by the electrolytic solution, jet polishing was unsuccessful. The only viable method of further thinning metal matrix composite specimens was by means of ion milling.

The concept of ion milling is to accelerate ionized atoms toward a target to knock off atoms from the specimen surface. The ion thinning process was performed with the Gatan 600 dual ion mill (Figure 1). Argon gas was used as the ion source. The following five key factors that must be taken into consideration when ion milling are:

1. incidence angle of ion beam,
2. energy of argon ions,
3. gun current,
4. heating of the specimen, and
5. contamination.

Through experience gained from many ion milling experiments on various types of metal matrix composites (FP/Mg, FP/Al-Li, and Gr/Mg), some general operating principals were established. It was determined that the optimal incident angle of the ion beam was dependent on the fiber material characteristics. The optimal energy used to thin composites was dependent on the matrix material. It was concluded that the ion milling angle used to thin FP/ZE41A Mg without inducing etching effects was approximately 10° to 15° while the optimal ion energy used on FP/ZE41A Mg without causing ion damage was approximately 4 kV. The gun current settings were proportional to the

ion gun energy. A liquid nitrogen stage was used to maintain the specimen at low temperature during ion milling. A liquid nitrogen trap was installed near the diffusion pump to prevent excessive contamination. The total time of ion milling a 50 μm FP/ZE41A Mg to a 1000 \AA to 500 \AA composite thin foil was approximately 100 hours.

The FP/ZE41A Mg thin foils were examined under the JEOL 200CX TEMSCAN transmission electron microscope (Figure 2). All the specimens were imaged at 200 kV. Selective area diffraction patterns were obtained and analysed in order to identify unknown phases and to establish relative crystallographic orientations within a given microstructure.

Dislocation density measurements were performed by the method of line intersections⁵ utilizing the formula:

$$\Lambda = 2N/(Lt)$$

where Λ = dislocation density;

N = number of intersections with dislocations made by a random line with length L ;

L = length of a random line; and

t = specimen thickness.

N and L were determined on a large selection of random electron micrographs revealing the matrix dislocation networks. The specimen thickness was measured using convergent beam diffraction patterns.⁶

DISCUSSION AND RESULTS

Preliminary examination of the as-fabricated FP(α -Al₂O₃)/ZE41A magnesium revealed the three major areas of interest (Figure 3):

- a. the FP(α -Al₂O₃) fiber;
- b. the fiber/matrix interfacial reaction zone; and
- c. the ZE41A magnesium matrix.

Inspection of the FP(α -Al₂O₃) filament microstructure showed irregular polyhedral grains of alumina with an average grain size of approximately 0.50 μm (Figure 4). Small voids were occasionally found in individual grains. These voids may have originated during the filament fabrication process.

The interfacial zone of the as-fabricated FP/ZE41A Mg had a width of approximately 0.25 μm from the alumina interface (Figure 3). This reaction zone consisted of large particles (0.50 μm in diameter) and a thin film of reaction product (Figure 5). Selective area electron diffraction was performed on the larger interfacial particles which were subsequently identified as magnesium oxide (Figure 6). Upon heat treatment at 450 °C for 95 hours, the larger magnesium oxide particles decreased in size to smaller precipitates whereas the overall reaction zone grew from 0.25 μm to 2 μm in width (Figure 7). Through selective area electron diffraction, this reaction zone was identified as consisting of fine precipitates of MgAl₂O₄ spinel (Figure 8). Since the reaction zone grew from the original thin film at the interface

⁵ HIRSH, P. B., HOWILL, A., NICHOLSON, R. B., PASHLEY, D. W., and WHELAN, M. H. *Electron Microscopy of Thin Crystals*. Butterworths, Washington, 1965.

⁶ ALLEN, S. M. *Foil Thickness Measurements from Convergent Beam Diffraction Pattern*. *Philosophical Magazine A*, v. 43, no. 2, 1981, p. 325-335.

of the as-fabricated composite, it would be reasonable to assume that the initial reaction thin film was also the spinel $MgAl_2O_4$. Therefore, during the composite consolidation process in which molten magnesium was in contact with the alumina fiber surface, free oxygen on the filament surface reacted with the magnesium to form magnesium oxide. Subsequently, the magnesium oxide reacted with the alumina to form the spinel $MgAl_2O_4$. The interfacial diffraction ring patterns indicated that the spinel had an ultrafine microstructure. Such an ultrafine structure at the interface may be beneficial in transferring loads from the low-strength, low-modulus matrix material to the high-strength, high-modulus filament.⁷ The reaction zone may have also served as a thermal stress relieving gradient. However, the large magnesium oxide particles at the interface may be detrimentally acting as stress concentration points for initiating interfacial failure.

In addition to the interfacial reaction zone encircling the fiber circumference, a concentric area of relatively small magnesium grains surrounded the reaction zone (Figure 9). These grains of 0.50 to 1 μm in width formed at the reaction zone occurred as narrow overlapping grains (Figure 9) or exhibited a spherical shape (Figure 10). These structures most likely resulted from the initial contact between the hot molten metal and the relatively cool alumina surface. The area may have also interacted beneficially with the interfacial reaction zone to promote load transfer and to relieve thermally induced stresses.

Examination of the magnesium matrix away from the alumina fiber showed dislocations and precipitates in the microstructure (Figure 11). The dislocation density of the as-fabricated composite matrix was approximately 10^{10} cm^{-2} , typically that of a cold-worked structure.⁸ The predominant precipitates found in the as-fabricated FP/ZE41A Mg were in the form of short rods (Figure 12). These rod-like precipitates lay parallel to the c-axis of the the magnesium matrix and were identified as the coherent transition phase $MgZn'$ with a structure similar to the laves phase $MgZn_2$ (Figure 13). The very same precipitate has been reported by Clark⁸ in Mg 5% Zn alloy aged from 149°C to 260°C. Spherical precipitates were also observed along twin boundaries (Figure 14). These precipitates have been indentified as the equilibrium phase $MgZn$ (Figure 15).

Upon heat treatment of FP/ZE41A Mg at 450°C for 95 hours, polygonization occurred within the matrix material (Figure 16). Dislocations glided to form sub-grain boundaries resulting in strain-free sub-grains. Concurrently, the rod-like $MgZn'$ precipitates had disappeared and a spherical precipitate had formed (Figure 17). This idiomorphic precipitate may also be the equilibrium $MgZn$ phase formed after the loss of coherency of the $MgZn'$ as reported by Levitt.⁹

In summary, the microstructure observed in FP/ZE41A Mg indicated that the fine grain spinel $MgAl_2O_4$ at the interfacial reaction zone and the spherical-like structures encircling the filament may be the beneficial elements in assisting load transfer and in relieving thermally induced stresses at the interface. The large magnesium oxide particles at the interface may be a potential source of premature interfacial failure. However, heat treatment has shown that the MgO particle may be reduced to much finer and less detrimental sizes. The ZE41A Mg matrix is strengthened by $MgZn'$ and $MgZn$ precipitates.

7. HULL, D., and BACON, D. J. *Introduction to Dislocations*. Third Edition. Pergamon Press. New York, 1984.

8. CLARK, H. B. *Transmission Electron Microscopy Study of Age Hardening in a Mg-5% wt. Zn Alloy*. *Acta Metallurgica*, v. 13, December 1965.

9. LEVITT, A. P. *Whisker Technology*. Wiley-Interscience, New York, 1970.

In conclusion, this microstructural analysis of the fiber/matrix interface in the FP/ZE41A composite indicates that this interface consists of a thin layer ($\sim 0.25 \mu\text{m}$) of MgAl_2O_4 spinel and relatively large magnesium oxide (MgO) ($\sim 0.5 \mu\text{m}$) particles. Heat treatment at 450°C for 95 hours caused the spinel interfacial layer to grow ($\sim 2 \mu\text{m}$) at the expense of reducing the MgO particle size.

CONCLUSION

1. The as-fabricated $\text{FP}(\alpha\text{-Al}_2\text{O}_3)/\text{ZE41A Mg}$ composite consisted of the following microstructural features:
 - a. Polycrystalline alumina fibers with a $0.50\text{-}\mu\text{m}$ diameter grain size;
 - b. The interfacial reaction zone between the fiber and matrix material consisted of magnesium oxide particles and a thin layer of fine MgAl_2O_4 spinel precipitates; and
 - c. The matrix material consisted of fine dispersion of rod-like MgZn' precipitates, dislocations, and MgZn precipitates along twin boundaries.
2. The composite interfacial reaction zone grew with aging at 450°C from $0.25 \mu\text{m}$ in width in the as-fabricated condition to $1\text{-}2 \mu\text{m}$ in width.
3. Heat treatment at 450°C reduced the magnesium oxide interfacial particles in size.
4. Polygonization of the magnesium matrix occurred with aging at 450°C .
5. Equiaxed precipitates likely to be the equilibrium phase MgZn were found in the $450^\circ\text{C}/95 \text{ hr}$ annealed specimen with the MgZn' phase missing.

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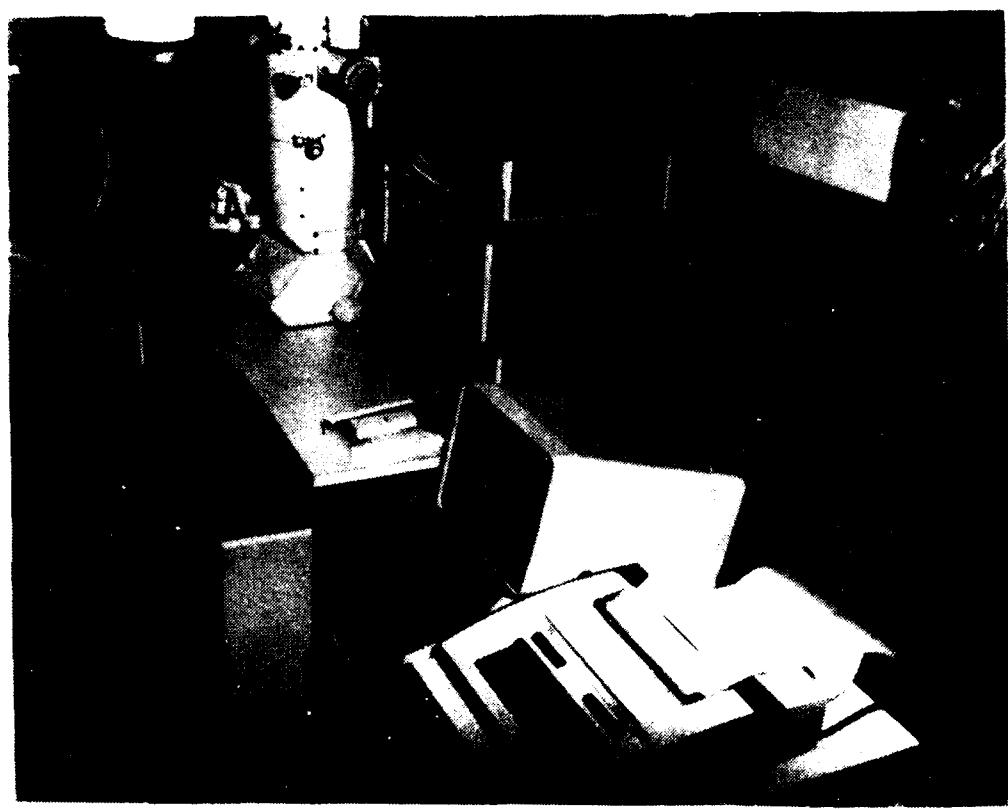


Figure 1. Gatan 600 dual ion mill.

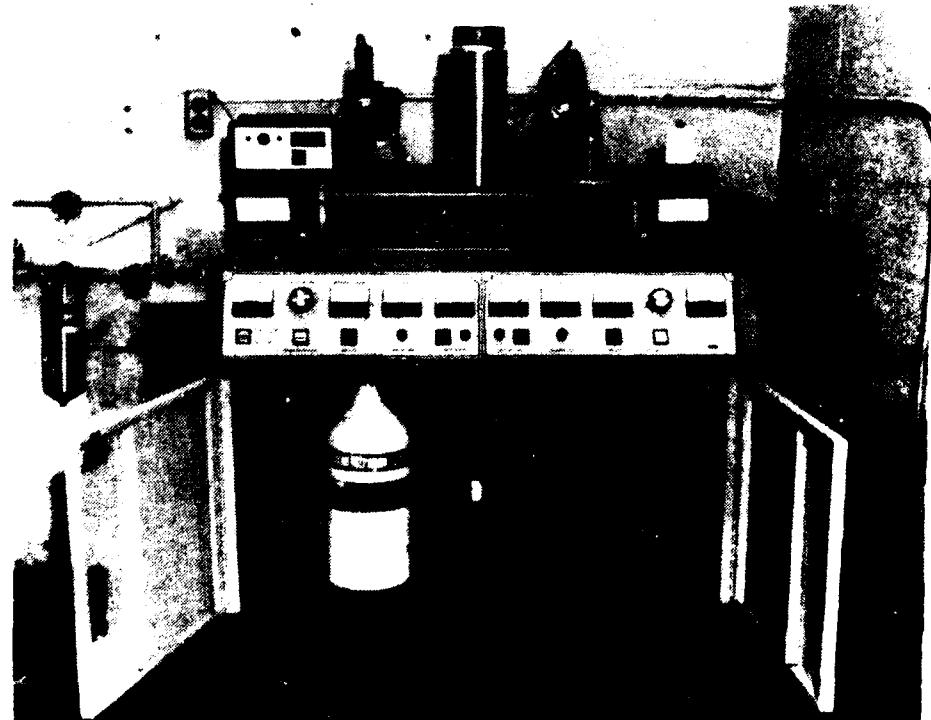


Figure 2. JEOL 200CX TEMSCAN.



Figure 3. Transmission electron micrograph of as-fabricated FP(α -alumina)/ZE41A Mg revealing (A) the α -alumina fiber, (B) the interfacial reaction zone, and (C) the magnesium matrix.



Figure 4. Transmission electron micrograph of FP(α -alumina) filament.

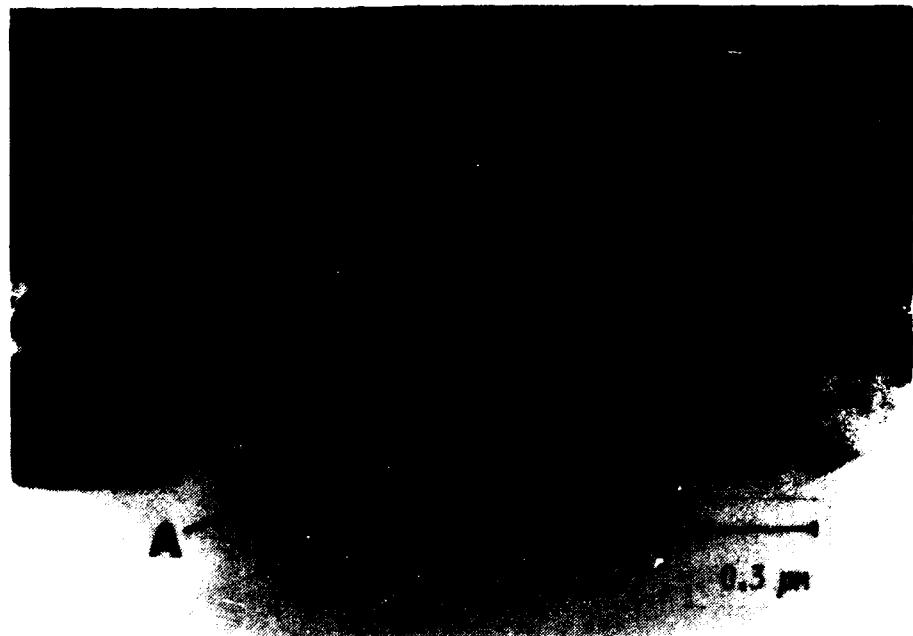


Figure 5. Microstructural features of the alumina/magnesium interface showing (A) the large magnesium oxide particles and (B) the thin reaction zone of MgAl_2O_4 spinel.

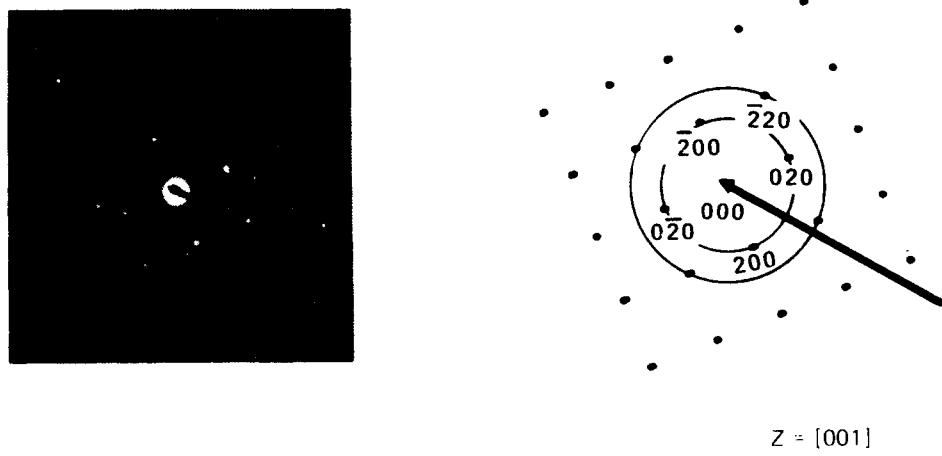


Figure 6. Electron diffraction pattern of the interfacial magnesium oxide particle.

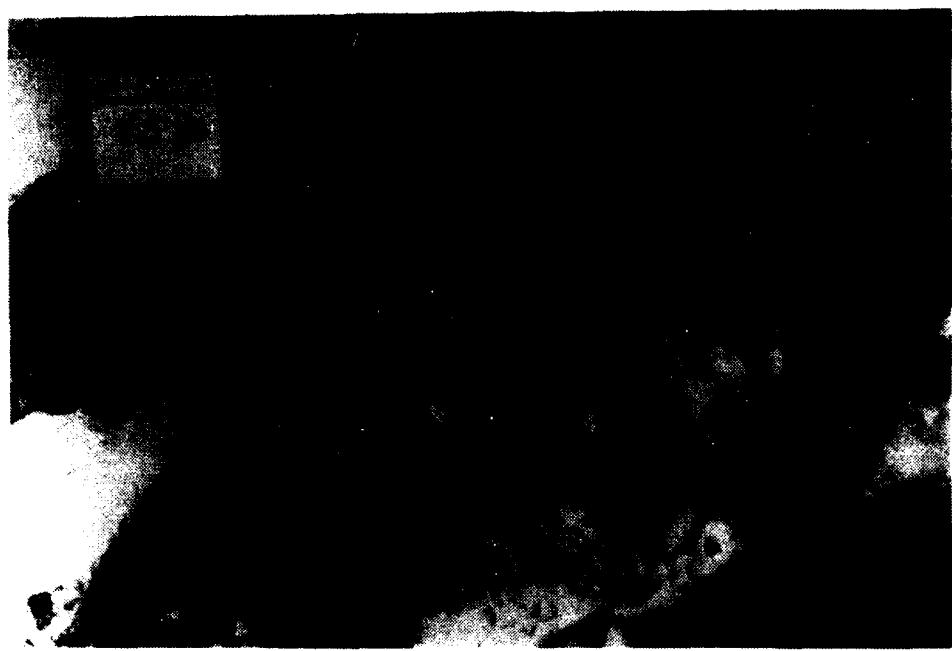


Figure 7. Transmission electron micrograph of 450°C/95 hr heat treated FP(α -alumina)/ZE41A magnesium interface showing the enlarged reaction zone (shown at "A").

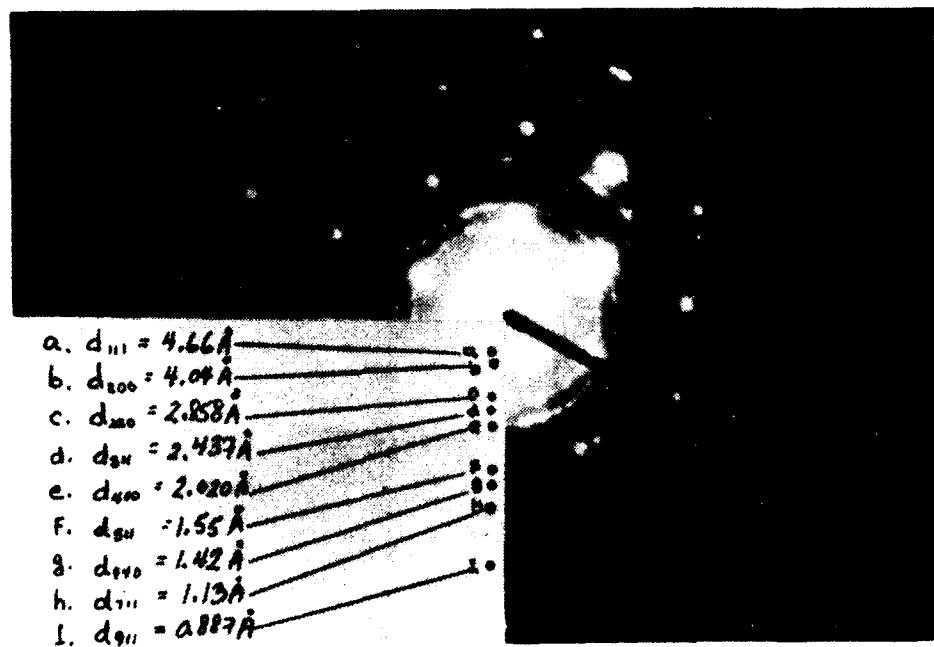


Figure 8. Electron reflection from the textured MgAl_2O_4 spinel phase formed at the alumina/magnesium interface.

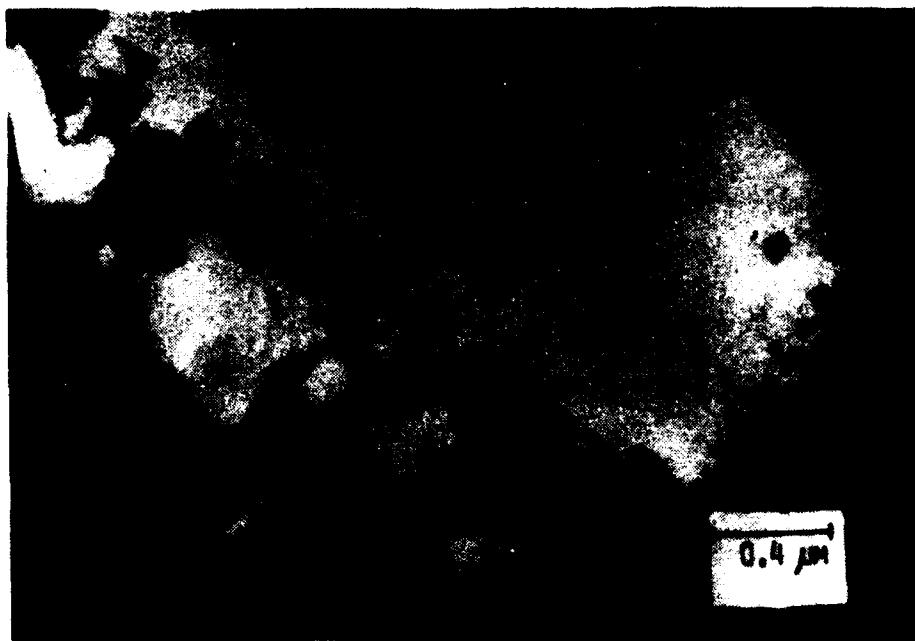


Figure 9. Transmission electron micrograph of a lapping grain formed around the reaction zone (shown at "A").



Figure 10. Transmission electron micrograph of spherical structures formed at the reaction zone (shown at "A").

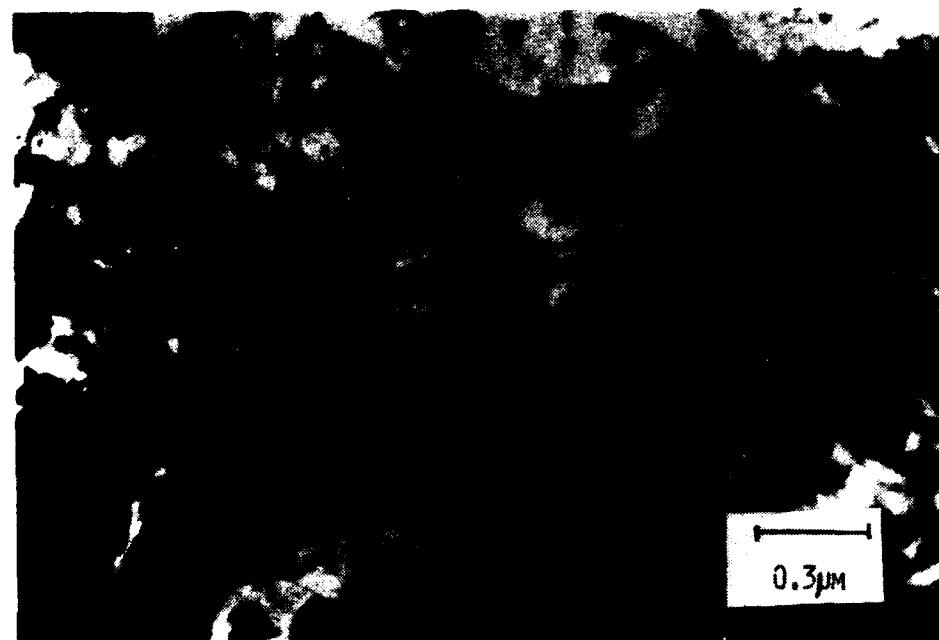


Figure 11. Microstructural features in the ZE41A magnesium matrix showing networks of dislocations and precipitates.

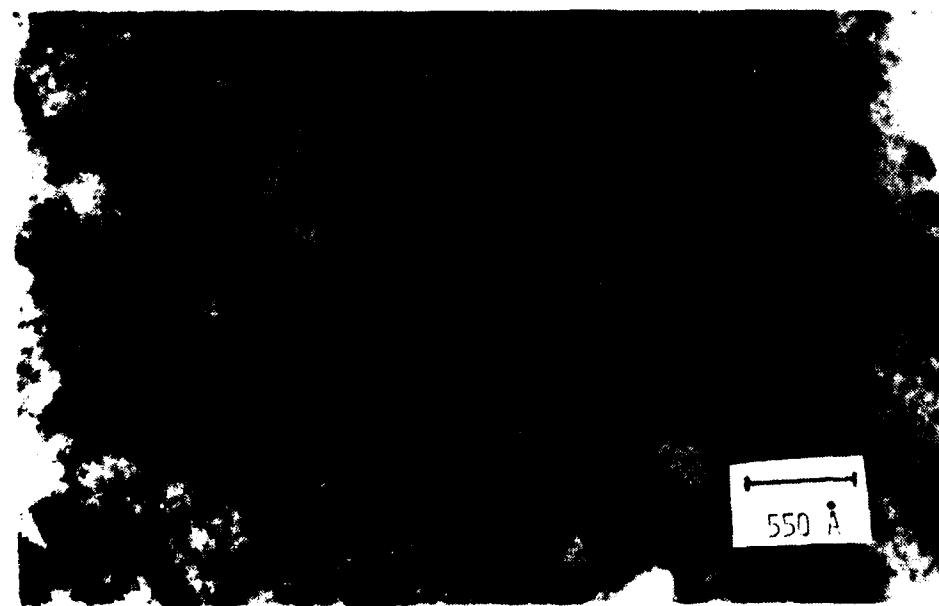


Figure 12. Higher magnification of the rod-like precipitates found in the ZE41A magnesium matrix.

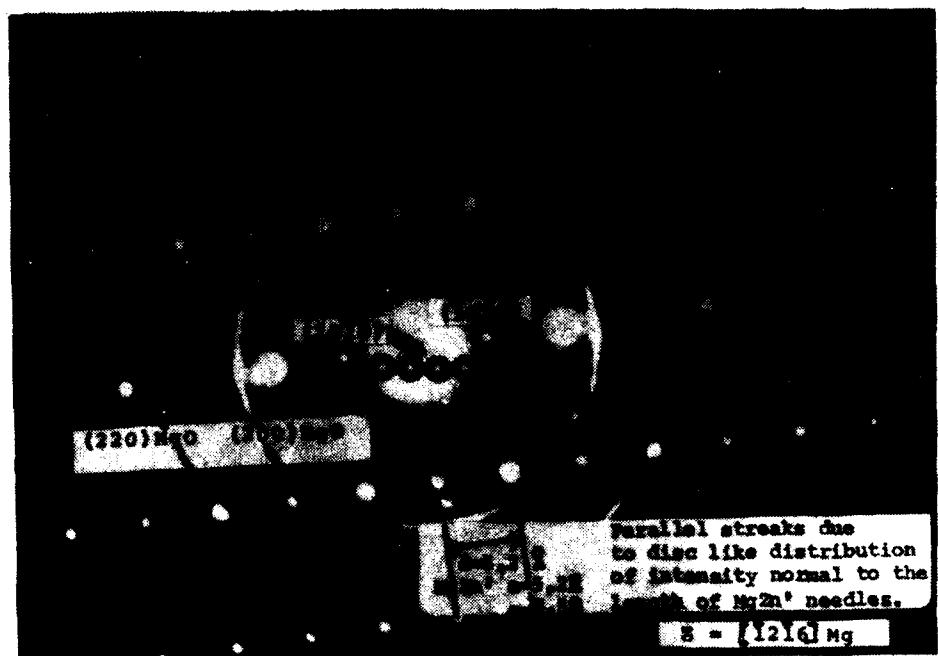


Figure 13. Electron diffraction pattern of MgZn' precipitates showing the reciprocal lattice planes parallel to the basal plane of the the magnesium.

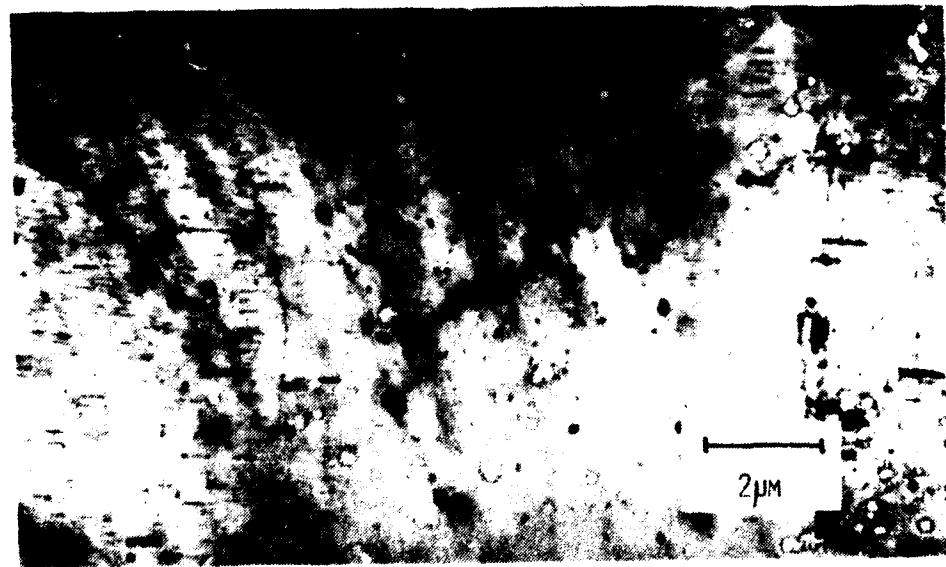
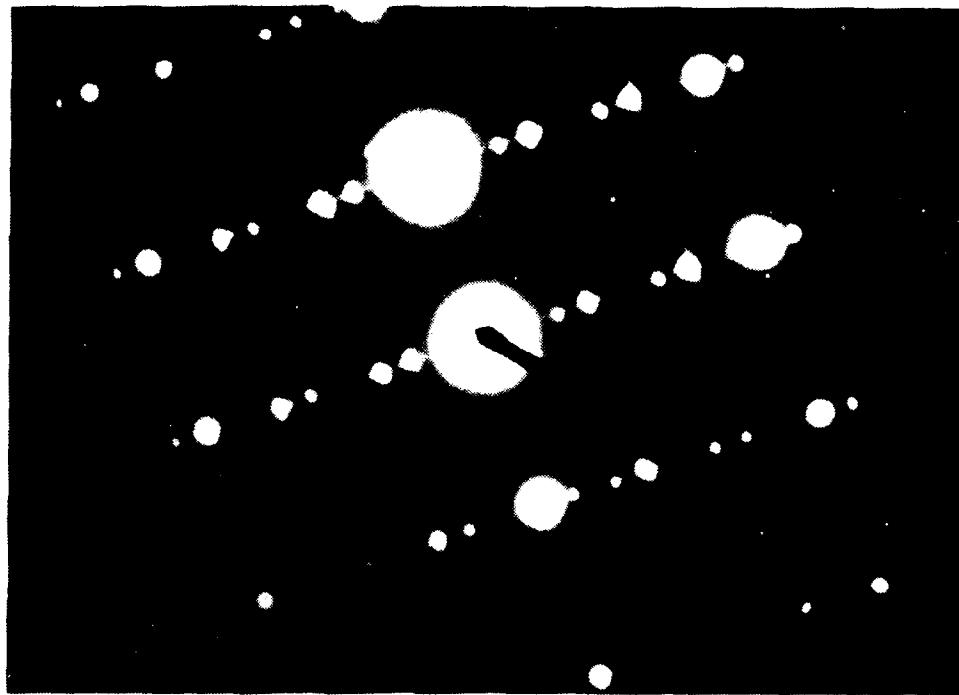


Figure 14. Transmission electron micrograph of precipitates along twin boundaries.



○ Mg ZMg = $[01\bar{1}\bar{0}]$

✗ MgZn

• Double Diffraction

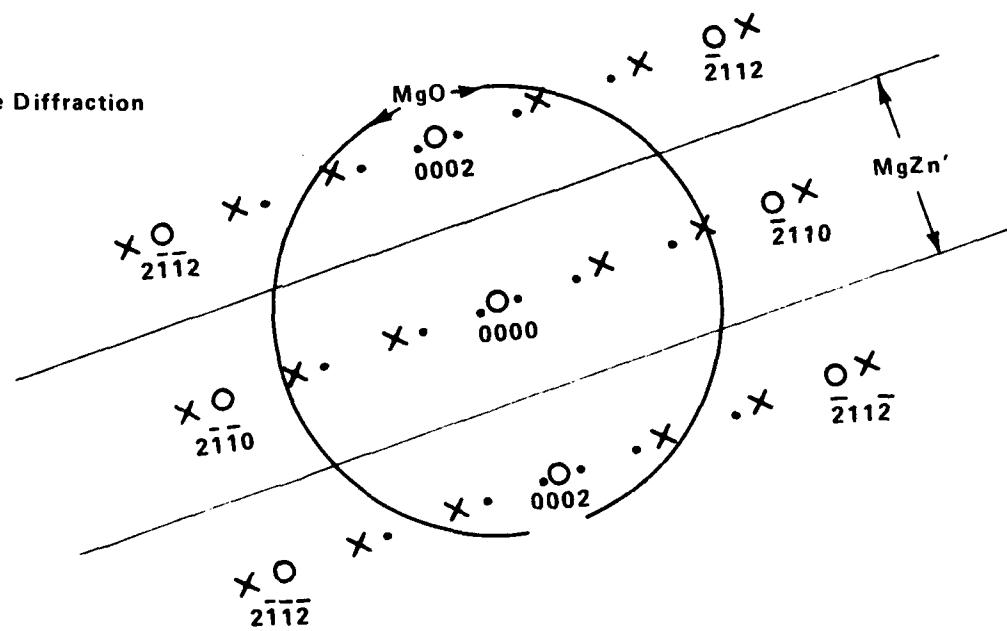


Figure 15. Electron diffraction pattern showing MgZn reflections.



Figure 16. Transmission electron micrograph of 450°C/95 hr heat treated FP(α -alumina)/ZE41A magnesium composite showing the polygonization of the matrix material.



Figure 17. Transmission electron micrograph of equiaxed precipitates formed in 450°C/95 hr heat treated FP(α -alumina)/ZE41A magnesium matrix.

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U.S. Army Materials Technology Laboratory,
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TRANSMISSION ELECTRON MICROSCOPY STUDY OF
ALUMINA FIBER-REINFORCED MAGNESIUM
COMPOSITE - Ernest Sung Chor Chin

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A detailed microstructural analysis of alumina (f) fiber-reinforced ZE41A magnesium composite was performed by means of transmission electron microscopy (TEM). Fifty-five volume percent f fiber-reinforced ZE41A magnesium composite thin foils were prepared by methods of mechanical polishing and ion thinning. Three major areas of interest in the composite microstructure were studied: the filaments, the interface (fiber/matrix reaction zone), and the matrix. In addition, the effects of heat treatments at 450°C on the composite microstructures were investigated. It was found that the alumina/magnesium interface consisted of a thin layer of MgAl₂O₄ spinel and relatively large magnesium oxide (MgO) particles. A concentric layer of magnesium alloy grains encircling the alumina filaments was also observed. Mg₂N₁ precipitates were also identified within the magnesium matrix microstructures. Upon heat treatment at 450°C for 95 hours, polygonization of the magnesium matrix occurred while the spinel interfacial layer grew with decreasing magnesium oxide particle size.

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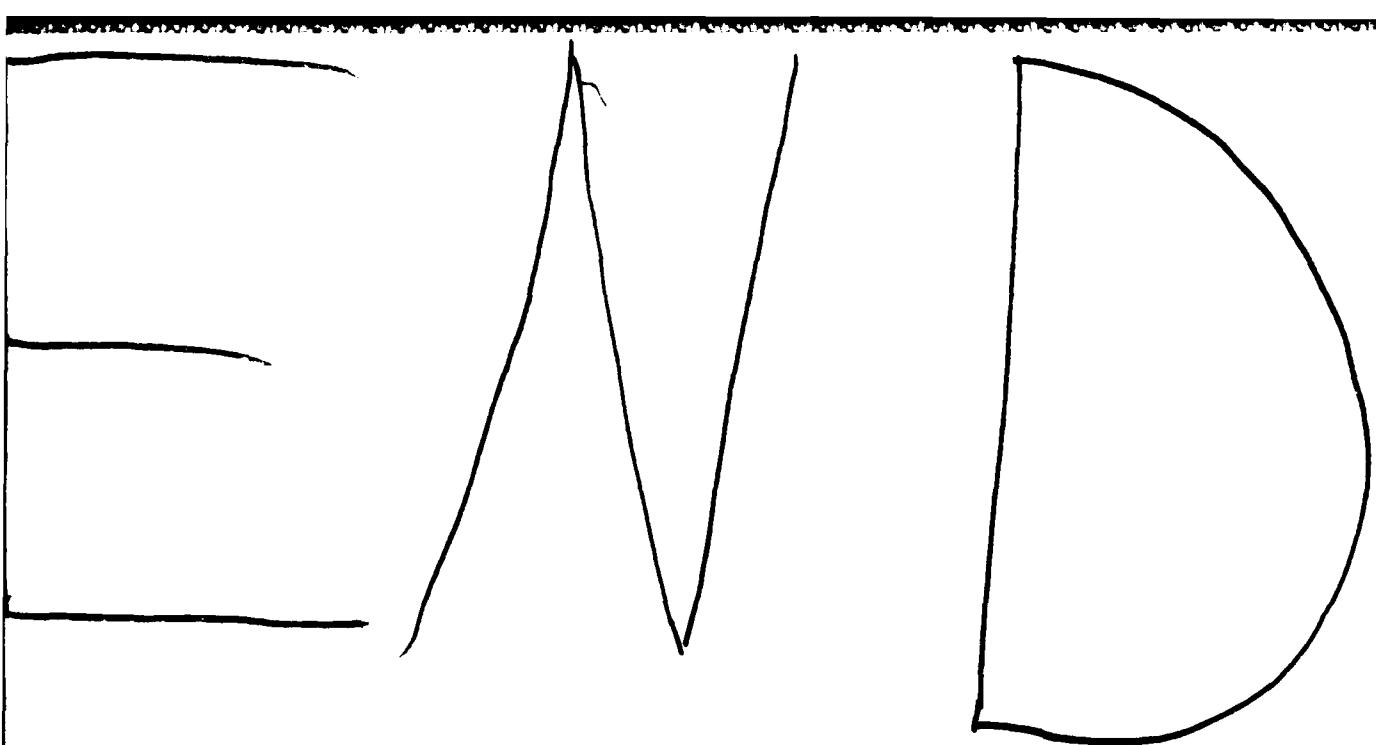
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